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Volumetric and compressibility studies on extractant-diluent system: effect of chain length

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ABSTRACT

The experimental densities ρ and sound velocities u for binary mixtures of TBP–dodecane have been measured at temperatures from 298.15 to 323.15 K and atmospheric pressure over the whole mole fraction range. From these data, the excess molar volume (V^E) and deviation in isentropic compressibility (Δk_s) have been calculated and fitted to Redlich–Kister polynomial equation. V^E and Δk_s values are positive for TBP–dodecane over the whole mole fraction range and at the above-mentioned temperatures. Excess thermal expansion coefficients (α^E) of a mixture of equimolar composition and deviations in molar volume of the components at infinite dilution have been calculated at all temperatures. The variations of these properties with composition and temperature of the binary mixture are discussed in terms of molecular interactions.

Keywords: Excess molar volume; Isentropic compressibility; Thermal expansion coefficient; TBP; Dodecane

1. Introduction

Solvent (liquid–liquid) extraction using organic solvents has been shown to be an efficient technique for separation and preconcentration of metals [1,2]. One of the most useful solvents, for investigating of this kind of reaction is tri-n-butyl phosphate (TBP). TBP is also known as solvating type of extractant. It has been widely used for industrial extraction of various types of organic compounds, metal ions including ones connected with the nuclear fuel cycle and inorganic acids from aqueous solutions [3–9]. Scientists have always emphasized on the importance of interactions involved during the distribution of inorganic species between water and various organic solvents during a solvent extraction process [10–12]. In these solute–solvent interactions, the complex-forming property of an organic molecule dissolved in a suitable organic solvent (diluent) is considered to be an important factor. This complex forming ability of the extractant with the solute will be influenced by the nature and extent of solute–extraction and extractant–diluent interaction. So, it is quite evident that knowledge of the intermolecular interactions among the diluent and extractant is of primary importance, since it is one of the factors which help to design the solvent extraction process with greater efficacy.

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Table 1
Comparison of experimentally measured densities, ρ and ultrasonic speeds, u of pure components with literature values

Component	T/K	ρ/kgm^{-3}		u/ms^{-1}	
		Exp	Lit	Exp	Lit
Doubly distilled water	298.15	997.041	997.043[17]	1497.57	1497.00[18]
Tributyl phosphate	298.15	973.849	972.70[19]	1272.48	–
			972.49[20]		–
n-Dodecane	293.15	748.963	749.50[19]	1298.06	–
	298.15	745.346	–	1278.65	–

Thermodynamic properties like excess molar volume, excess molar enthalpy, and excess isentropic compressibility improve our understanding about the behavior of liquid mixtures. Density and sound velocity of pure and mixed solvents may help in shedding light on the aspect of intermolecular interactions, since these are affected by differences in interactions between like as well as unlike molecules, which in turn influence the molecular arrangement in various liquid mixtures. Molecular arrangement influences the interstitial accommodation of components among each other. Since volumetric properties depend on solute–solute, solvent–solvent, and solute–solvent interactions and on structural effects arising due to differences in molar volume and free volume between components present in solution, volumetric data can be used to test molecular theories and/or models of solution for understanding the molecular interactions between components.

Since reports on the interaction of extractants with diluents from thermodynamic point of view are very few [13–15], we have initiated some systematic studies on evaluation of excess thermodynamic parameters for systems used in the solvent extraction technique by choosing different types of combinations amongst the components [16]. In this work, we have prepared solutions containing different proportions of TBP mixed with dodecane and derived the molar excess volume and excess isentropic compressibility on mixing from the density and sound velocity of the pure and mixed binary solvents. The trend in molar excess volume and isentropic compressibility shown in case of TBP–dodecane mixture is compared with that of TBP–hexane mixture already published by the authors [16]. We have tried to draw some conclusion on how the nature of interaction changes between the extractant and solvent, particularly, when the chain length of the diluent changes.

2. Experimental

TBP was purchased from E. Merck, Germany (purity > 99%) and dodecane was purchased from S.D. Fine Chemicals Ltd. (purities > 99%) and used as received. A set of liquid mixtures were prepared, with the mole fraction of TBP varying from 0.1 to 0.9 in each case by weight, using a Mettler balance with an accuracy of ± 0.0001 g. The density and sound velocity of the solutions were measured in a temperature range of 298.15–323.15 K (with temperature measuring accuracy of ± 0.001 ℃) by an automated density and sound velocity meter DSA 5,000 M having a density measurement accuracy of ± 0.000005 gm/cm^3 and sound velocity measurement accuracy of 0.5 m/s. The density meter was previously calibrated using doubly distilled water. The accuracies of density and ultrasonic speed measurements were ascertained by comparing the experimental values for pure liquids with the literature (Table 1). It is seen that although the compounds have been used as received, the deviations in density and sound velocity values from the literature data are within 1%. The slightly higher value of the density of TBP might be due to traces of impurities like water.

3. Results and discussion

From the experimental ρ and u data, the excess molar volume, V^{E}, and deviations in isentropic compressibility, Δk_{s} have been calculated using the following relations:

$$V^E = (x_1M_1 + x_2M_2)/\rho - (x_1M_1/\rho_1) - (x_2M_2/\rho_2) \qquad (1)$$

$$k_{\mathrm{s}} = 1/(u^2\rho) \qquad (2)$$

$$\Delta k_{\mathrm{s}} = k_{\mathrm{s}} - (\varphi_1 k_{s,1} + \varphi_2 k_{s,2}) \qquad (3)$$

where φ is the volume fraction, M is the molar mass, and k_{s} is the isentropic compressibility. 1 and 2 denote pure components TBP and dodecane, respectively. The experimental values of densities, ρ and ultrasonic sound velocities, u of pure TBP and dodecane and those of their binary mixtures, the calculated values of excess molar volume (V^E), and deviations in isentropic compressibility (Δk_s) over the entire composition range expressed by mole fractions x_1 of TBP and at temperatures 298.15, 303.15, 308.15, 313.15, 318.15, and 323.15 K, respectively, are listed in Table 2.

The variations of V^E and Δk_s with mole fraction x_1 of TBP at two temperatures for the binary mixture of TBP–dodecane and TBP–hexane (for comparison) are plotted in Figs. 1 and 2, respectively. The excess functions are found to be positive in case of TBP–dodecane and negative in case of TBP–hexane system [16]. The structure of dodecane somewhat resembles that of a cylinder with the axis made up of the main –C—C— backbone and a TBP molecule can be visualized as an "inverted umbrella." The structures of these two molecules are thus distinctly different from each other, although the molar volume of dodecane (2.2854×10^{-4} m^3) is comparable to that of TBP (2.7347×10^{-4} m^3). This difference in structure results in poor CMO effect among the two [21] and this energetically destabilizes the TBP–dodecane moeity. Due to similar molar volume of the two with different structure, the steric repulsion between the two is considerably high so that the dipole–induced dipole force of attraction (which varies inversely as the sixth power of the distance between them) gets reduced in comparison to hexane. Thus, the reduction of attractive force between TBP and dodecane together with the factor of steric repulsion play a dominating role in the entire composition range, thereby increasing the interspace between the molecules causing a positive deviation from ideality for volume and isentropic compressibility. In case of TBP–hexane binary mixtures, the molar volume of hexane (1.3153×10^{-4} m^3) being much smaller than that of TBP (2.7347×10^{-4} m^3) gets incorporated into the tetrahedral portion of the TBP molecule that is surrounded by the alkoxy groups when TBP acts as the solvent, thus decreasing the interspace between the molecules. This gets reflected in the negative excess volume and negative deviations of isentropic compressibility. In the situation where TBP finds itself surrounded by hexane molecules, the interactions between these two molecules comprise of a dominant steric part, which allows favorable accommodation of hexane into the "tributyl umbrella" of TBP, followed by stabilizing dipole-induced dipole interactions between the two.

Use of experimental data in practical applications necessitates interpolation or extrapolation that is possible only through proper mathematical representation. The experimentally obtained excess functions can be best represented as power series in such a way that there are additive higher-order terms, which serve as corrections to the terms of lower order. Since excess functions are zero for $x_1 = 0$ or 1, each term should contain a factor x_1x_2 and also it is desirable to develop the series in terms of a variable which is symmetric with respect to the two components and the simplest variable of this kind is (x_1–x_2). The most useful development is of the following type [22]:

$$Y^E = X_1X_2 \sum_{i=1-5} A_{i-1}(X_1 - X_2)^{i-1} \tag{4}$$

where Y^E stands for the excess function. The coefficients A_i of Eq. (4) are parameters that can be adjusted for a better fit of the excess functions and were calculated by the method of multiple regression using Origin Software Version 6.1. These parameters have theoretical significance, since they can be equated to the respective apparent molar quantities over the whole composition range and their extrapolation to $x_1 = 0$ or 1 gives the partial molar quantities under the condition of infinite dilution which gives a measure of solute–solvent interactions [23]. The values of V^E and Δk_s of the binary mixtures, at each investigated temperature were fitted to a Redlich–Kister type polynomial equation (Eq. (4)) and the values of the coefficients are tabulated (Table 3). Using these values of the coefficients, the calculated values of the excess functions (V^E and Δk_s) were plotted in Figs. 1 and 2 as curved lines.

The excess molar volume (V^E) of TBP–dodecane system at different temperatures is plotted in Fig. 3 and it shows a slight positive increment in V^E values with increasing temperatures from 298.15 to 323.15 K. For comparison, a similar plot of TBP–hexane system [16] at near equimolar composition is also plotted in Fig. 3 at the same temperature, while it shows the opposite result to that of TBP–dodecane system. Here, with increasing the temperature, the randomness of the motion of both TBP and dodecane molecules favor a relatively closer approach between them, whereas the effect of temperature enhancement was explained elsewhere [16] as the better packing among TBP monomers and hexane molecules.

The excess thermal expansion coefficients of the binary mixtures were calculated at the experimental temperatures using the following relations [24]:

$$\alpha = -(1/\rho)(\partial\rho/\partial T) \tag{5}$$

$$\alpha^E = [V_{\mathrm{id}}^{-1}(\partial V^E/\partial T) - \alpha V^E] \tag{6}$$

where the molar volume for ideal mixture V^{id} is equal to (x_1V_1+x_2V_2). These values are presented in Table 4. It is seen that α^E and ($\partial V^E/\partial T$) have opposite signs

Table 2
Densities (ρ), sound velocities (u), excess molar volume (V^E), and deviations in isentropic compressibility (Δk_s) for (TBP (1) + alkane(2)) binary mixtures at different temperatures

Temp/K	x_{TBP}	ρ (kgm^{-3})	u (ms^{-1})	V^E ($\times 10^6$) (m^{3}mol^{-1})	Δk_s ($\times 10^{11}$) (m^{2}N^{-1})
(TBP(1) + n-Dodecane(2))					
298.15	0.0000	745.346	1278.65	0	0
	0.0500	758.641	1272.56	0.07404	0.045406
	0.1500	784.042	1263.79	0.34314	0.105799
	0.3500	831.572	1253.82	0.97309	0.176025
	0.5500	876.673	1250.97	1.26641	0.192207
	0.7500	919.501	1253.54	1.30148	0.174749
	0.8500	940.867	1256.68	1.02468	0.149756
	0.9500	962.301	1261.66	0.54143	0.108348
	1.0000	973.849	1272.48	0	0
303.15	0.0000	741.707	1259.45	0	0
	0.0500	754.950	1253.55	0.07539	0.047012
	0.1500	780.241	1245.18	0.35152	0.108658
	0.3500	827.613	1235.7	0.98248	0.181797
	0.5500	872.580	1233.28	1.27227	0.198465
	0.7500	915.311	1236.25	1.2956	0.179834
	0.8500	936.636	1239.57	1.00979	0.153730
	0.9500	958.020	1244.6	0.51930	0.111992
	1.0000	969.444	1255.46	0	0
308.15	0.0000	738.064	1240.34	0	0
	0.0500	751.254	1234.62	0.07711	0.048818
	0.1500	776.439	1226.42	0.35924	0.114842
	0.3500	823.641	1217.61	0.99499	0.188567
	0.5500	868.484	1215.65	1.27833	0.205180
	0.7500	911.121	1219.05	1.28902	0.184869
	0.8500	932.404	1222.46	0.99443	0.158659
	0.9500	953.740	1227.64	0.49612	0.115449
	1.0000	965.038	1238.51	0	0
313.15	0.0000	734.418	1221.43	0	0
	0.0500	747.554	1215.85	0.07904	0.05121
	0.1500	772.630	1207.92	0.36792	0.1203
	0.3500	819.667	1199.65	1.00646	0.196
	0.5500	864.380	1198.14	1.28449	0.21239
	0.7500	906.924	1201.89	1.28148	0.191
	0.8500	928.167	1205.51	0.97712	0.16307
	0.9500	949.453	1210.85	0.47119	0.11812
	1.0000	960.620	1221.65	0	0

(*Continued*)

Table 2
(*Continued*)

Temp/*K*	x_{TBP}	ρ (kgm^{-3})	*u* (ms^{-1})	V^E (× 10^6) (m^{3}mol^{-1})	Δk_s (× 10^{11}) (m^{2}N^{-1})
318.15	0.0000	730.766	1202.66	0	0
	0.0500	743.845	1197.25	0.08184	0.05328
	0.1500	768.815	1189.59	0.37643	0.12551
	0.3500	815.685	1181.81	1.01797	0.20392
	0.5500	860.276	1180.73	1.28776	0.22017
	0.7500	902.724	1184.9	1.2713	0.19666
	0.8500	923.928	1188.7	0.95651	0.16729
	0.9500	945.164	1194.18	0.44266	0.12076
	1.0000	956.189	1204.91	0	0
323.15	0.0000	727.105	1184.06	0	0
	0.0500	740.131	1178.82	0.08347	0.05535
	0.1500	764.995	1171.37	0.38402	0.13177
	0.3500	811.697	1164.12	1.0292	0.21219
	0.5500	856.162	1163.49	1.2921	0.22786
	0.7500	898.518	1168.04	1.26106	0.20252
	0.8500	919.684	1172.01	0.93553	0.1717
	0.9500	940.874	1177.62	0.41262	0.12358
	1.0000	951.754	1188.29	0	0

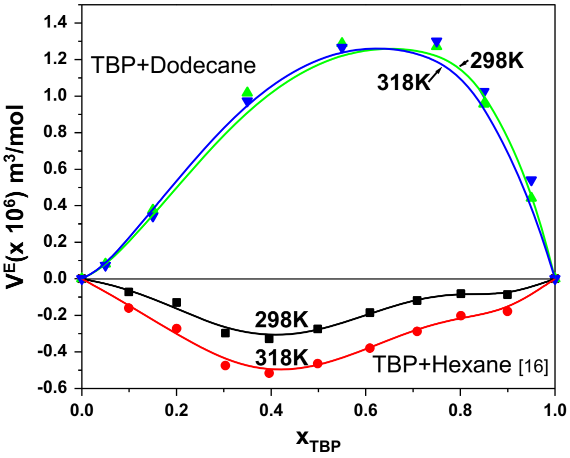

Fig. 1. Excess molar volumes of binary liquid mixtures of TBP + Hexane/Dodecane at two temperatures as a function of TBP mole fraction, x_1. (■,●) Experimental data for TBP–hexane at 298.15 K and 318.15 K respectively [16]. (▲,▼)-Experimental data for TBP–dodecane at 298.15 and 318.15 K, respectively. The curved lines indicate the theoretical curves obtained from the Redlich–Kister equation (Eq. (4)).

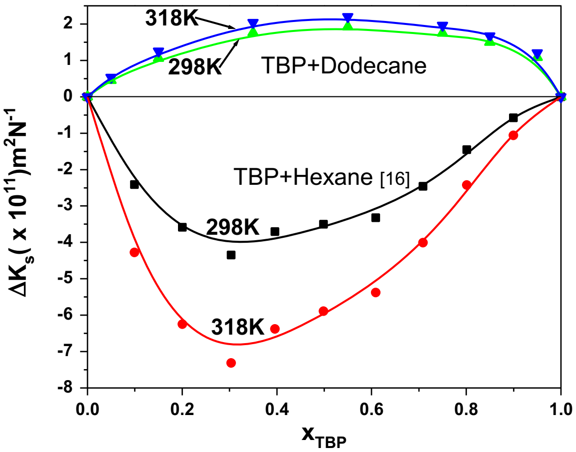

Fig. 2. Deviations in isentropic compressibilities of binary liquid mixtures of TBP + Hexane/Dodecane at two temperatures as a function of TBP mole fraction, x_1. (■,●) Experimental data for TBP–hexane at 298.15 K and 318.15 K respectively [16]. (▲,▼)-Experimental data for TBP–dodecane at 298.15 and 318.15 K, respectively. The curved lines indicate the theoretical curves obtained from the Redlich–Kister equation (Eq. (4)).

which indicate that for both these mixtures, V^E seems to be the dominating parameter. At 298.15 K and equi-molar composition, the percentage increase in the excess volume (0.25%) for TBP–dodecane system is much larger than the corresponding decrease in case of TBP–hexane mixture (0.07%) which leads to negative values of α^E for TBP–dodecane mixture and positive values for TBP-hexane.

The deviations in V^∞ at infinite dilution, ΔV_1 and ΔV_2 for TBP and the liquid alkane have been calculated (Table 5) from the Redlich–Kister polynomial equations using the following relations:

Table 3
Coefficients A_i of Redlich–Kister equation for the binary mixture at different temperatures

T/K	A_0	A_1	A_2	A_3	A_4
TBP + Dodecane/V^E *(* $\times 10^6$ m^3/mol*)*					
298.15	4.90698	2.25135	0.90461	3.6032	0.58244
303.15	4.9307	2.23627	1.02181	3.32736	0.10707
308.15	4.95915	2.19882	1.13630	3.08335	−0.39449
313.15	4.98637	2.17046	1.24958	2.79176	−0.90962
318.15	5.00413	2.13135	1.39716	2.47169	−1.50636
323.15	5.02486	2.10098	1.5396	2.137	−2.13383
TBP + Dodecane/Δk_s *(*$\times 10^{11}$ m^2/N*)*					
298.15	7.8345	−0.646737	−1.88499	7.97892	13.9109
303.15	8.09823	−0.692962	−2.20139	8.26608	14.6469
308.15	8.3707	−0.845075	−2.09073	8.53269	15.0402
313.15	8.66747	−0.908849	−1.97451	8.59381	15.2138
318.15	8.99196	−1.02143	−2.02997	8.75646	15.5297
323.15	9.31173	−1.17752	−1.89977	8.94492	15.6758

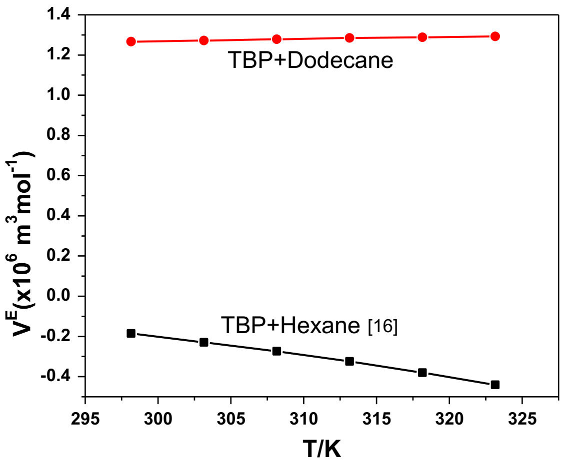

Fig. 3. Variation of excess molar volumes with temperature for the binary liquid mixtures at near equi-molar composition.

$$V_1^{\infty} = V_1^{\circ} + \sum_{i=1-5} A_{i-1}(-1)^{i-1} \tag{7}$$

$$V_2^{\infty} = V_2^{\circ} + \sum_{i=1-5} A_{i-1} \tag{8}$$

$$\Delta V_1 = \sum_{i=1-5} A_{i-1}(-1)^{i-1} \tag{9}$$

$$\Delta V_2 = \sum_{i=1-5} A_{i-1} \tag{10}$$

For TBP–dodecane mixture, the partial molar volumes at infinite dilution are higher than their corresponding molar volumes at each investigated temperature. This clearly suggests that there is an expansion in volume on mixing and also the extent of expansion decreases with increase of temperature. The reason for this is that dodecane, with a carbon chain much longer than the butyl group of the TBP molecule and having difference in shape and free volume with respect to TBP, cannot fully approach the latter at lower temperatures. This results in the expansion of the total volume of the TBP–dodecane system. When the temperature is increased, both randomness and free volume of the TBP molecule are increased and this favors closer approach of the dodecane molecule.

Besides deviations in isentropic compressibility, deviations in acoustic impedance and intermolecular free length derived from sound velocity through binary mixtures can also shed light on the nature of intermolecular interactions in these mixtures. The acoustic impedance, which is a measure of resistance offered by the liquid medium to the sound wave and intermolecular free length, which defines the distance between the surfaces of the molecules in a binary mixture, can be calculated using the following relations:

$$Z = u\rho \tag{11}$$

$$L_f = K/(u\rho^{1/2}) \tag{12}$$

Table 4
Experimental α^E and $(\partial V^E/\partial T)$ for the binary liquid mixtures at different temperatures and at near equimolar composition

System	T/K	α^E ($\times 10^4$ K^{-1})	$(\partial V^E/\partial T)$ ($\times 10^9$ m^{3}mol^{-1}K^{-1})
TBP + Hexane	298.15	2.6	−7.94
	303.15	3.0	−8.58
	308.15	3.5	−9.35
	313.15	4.0	−10.25
	318.15	4.6	−11.27
	323.15	5.2	−12.41
TBP + Dodecane	298.15	−11.8	1.25
	303.15	−11.9	1.22
	308.15	−12.0	1.14
	313.15	−12.2	1.00
	318.15	−12.3	0.818
	323.15	−12.4	0.580

Table 5
Values of deviations in partial molar volumes at infinite dilutions of TBP in dodecane at different temperatures using A_i parameters of Redlich–Kister equation

T/K	ΔV_1 ($\times 10^6$m^{3}mol^{-1})	ΔV_2 ($\times 10^6$m^{3}mol^{-1})	ΔK_1 ($\times 10^{11}$m^{2}N^{-1})	ΔK_2 ($\times 10^{11}$m^{2}N^{-1})
TBP(1) + Dodecane(2)				
298.15	0.53948	12.51073	12.52823	27.19260
303.15	0.49595	11.62321	12.97062	28.11685
308.15	0.41879	10.98313	13.63255	29.00778
313.15	0.36411	10.28855	14.22180	29.59172
318.15	0.29189	9.49797	14.75666	30.22672
323.15	0.19265	8.66861	15.32036	30.85516

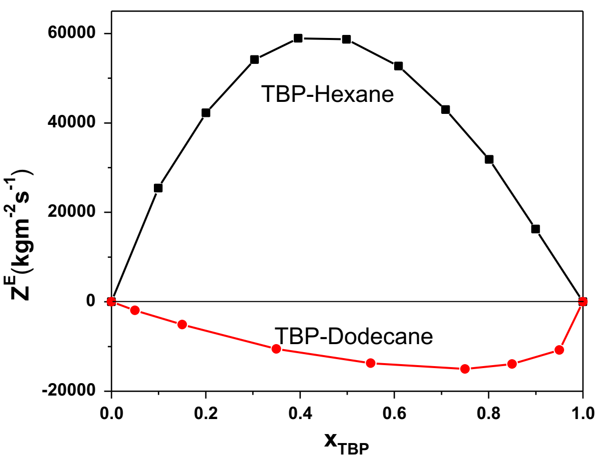

Fig. 4. Plot of deviations in acoustic impedance for binary mixtures of TBP with hexane and dodecane at 298.15 K.

where K denotes temperature dependent Jacobson's constant [25] given by $(93.875 + 0.375T) \times 10^{-8}$. The sign and extent of deviation of the functions from ideality are obtained from the excess functions which are calculated using the general equation:

$$X^E = X - (x_1X_1 + x_2X_2) \qquad (13)$$

where X represents Z or L_f and x_1 represents mole fraction of TBP and x_2 represent mole fraction of dodecane.

Deviation in acoustic impedance of TBP–dodecane mixture shows negative from ideal state (Fig. 4) and positive deviation in intermolecular free length (Fig. 5). Both these observations support the fact that on mixing of dodecane and TBP, there is an increase in interspace between these molecules due to unfavorable packing on account of mismatch of molecular structures. For comparison, the same data of TBP–hexane

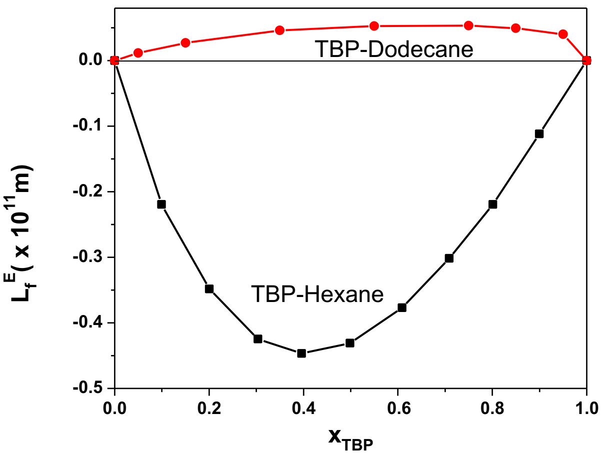

Fig. 5. Intermolecular free length for binary mixtures of TBP with hexane and dodecane at 298.15 K.

mixture [16] is also plotted in Figs. 4 and 5. Opposite trend in both figures of TBP–hexane system was explained in terms of favorable packing of hexane molecules into the TBP molecules. Similar trend is observed by Rajagopal et al. in their studies on interaction of 2-methyl-2-propanol with aliphatic nitriles [25].

4. Conclusions

In this paper, volumetric and acoustic data are reported for binary mixture (TBP–dodecane) at six different temperatures and compared with those of (TBP–hexane) mixture published earlier. Using these data, excess thermodynamic parameters such as deviations in isentropic compressibility, excess molar volume, excess intermolecular free length, and excess acoustic impedance have been calculated and interpreted in terms of intermolecular interactions among the components. These results are further complemented by calculation of partial molar volumes of the components under infinite dilution condition. It can be concluded that in both the systems, the solute and solvent molecules experience weak electrostatic attraction of dipole-induced dipole type. However, there is a marked difference in the magnitude of steric forces that are encountered by the solute and solvent molecules in the two systems, which is due to an appreciable difference in the molar volumes (due to difference in chain length) of hexane and dodecane. Negative deviations from ideality for volume, isentropic compressibility, and intermolecular free length (positive deviation in case of acoustic impedance) in case of TBP–hexane system and positive deviations for these parameters in TBP–dodecane system are, however, due to a compromise between the electrostatic and steric forces. This investigation can be correlated with extraction efficiency of TBP in hexane and dodecane [26] to get an idea of the extent to which solute–solvent interactions might influence the solvent extraction process.

Symbols

x_i — mole fraction of i th component
M_i — molar mass of i th component (gm mol^{-1})
ρ_i — density of pure i th component (kgm^{-3})
ρ — density of binary mixture (kgm^{-3})
φ_i — volume fraction of i th component
u — velocity of sound (ms^{-1})
k_s — isentropic compressibility of binary mixture (m^2N^{-1})
k_{si} — isentropic compressibility of the ith component (m^2N^{-1})
V^E — excess molar volume (m^3mol^{-1})
V_{id} — molar volume for ideal mixture (m^3mol^{-1})
V_i^{∞} — partial molar volume of ith component at infinite dilution (m^3mol^{-1})
ΔV_i — deviations in partial molar volume from that of pure ith component under infinite dilution (m^3mol^{-1})
α — thermal expansion coefficient (K^{-1})
Z — acoustic impedance ($kgm^{-2}s^{-1}$)
L_f — intermolecular free length (m)
K — Jacobson constant

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